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DETERMINATION OF PARTICLE SIZE OF POWDERS BY OPTICAL MICROSCOPE METHOD

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DETERMINATION OF PARTICLE SIZE OF POWDERS BY OPTICAL MICROSCOPE METHOD

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Indian Standard

DETERMINATION OF PARTICLE SIZE OF POWDERS BY OPTICAL MICROSCOPE METHOD

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 13 August 1969, after the draft finalized by the Sieves, Sieving and Other Sizing Methods Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Of the various methods employed for particle size determination, the optical microscope method is the only one in which direct observation is made of the size of the particles. Even this method has one drawback in that it has a tendency to measure the largest dimensions unless the particles are properly dispersed with random orientation. Also, the method is too much time consuming for extensive use between purchaser and supplier for determining conformity to specifications. But it may be used advantageously for two purposes, namely, (a) as a basis of calibration for faster methods such as those involving sedimentation and (b) in the determination of the particle size of routine samples, specially in the cases where particles are of such shape that they do not obey Stokes's law.

0.3 A number of methods are available for the determination of particle size in the range 1 - 1 000 microns. These are the microscope, elutriation, sedimentation and sieving. As no single method is applicable to the whole range of size 1 - 1 000 microns, it is necessary to combine the analysis of two or more different methods in order to establish the distribution in full. Moreover, the results obtained on particles between a given size range are not the same when determined by any two methods mentioned above. It is found that particle size determined by one method may be co-related to the size determined by another method by a certain multiplying factor. The correlation factors to be used in correlating particle size determined by various methods are given in Appendix A.

0.4 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

- B. S. 3406: Part 4: 1963 'Methods for determination of particle size of powders, Part 4 Optical microscope method' issued by the British Standards Institution.

ASTM Designation: E 20-62 T Tentative recommended practice for analysis by microscopical methods for particle size distribution of particulate substances of subsieve sizes.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS: 2-1960*.

1. SCOPE

1.1 This standard describes the optical microscope method for determining the particle size of powders which pass through a 75-micron IS Sieve IS: 460-1962† as far as the particle shape and refractive index will permit. The method of measurement is so arranged that the size analyses of the particles, in microns, are expressed in the form of a consecutive $\sqrt{2}$ series, namely 75, 53, 37, 27, 19, 13, 9.4, 6.6, 4.7, 3.3, 2.3, 1.7, 1.2, 0.8, 0.6‡.

1.2 The size distribution is expressed either by number or by volume or weight and in terms of the diameters of circles having the same projected areas as the particles. The method is normally used for particles having projected diameters in the range 0.6-75 microns but the upper size limit can be extended to measure particles up to 150 microns (see 4.3).

2. TERMINOLOGY

2.0 For the purpose of this standard, the following terms and definitions and that given in IS: 4124-1967§ shall apply.

2.1 Laboratory Sample—The portion of the gross sample which is delivered to the laboratory for determination of particle size distribution.

2.2 Analysis Sample—The portion of the laboratory sample which is used in the size analysis apparatus.

2.3 Intermediate Sample—The portion of the laboratory sample which includes the analysis sample.

3. OUTLINE OF METHOD

3.1 A representative sample of the powder to be sized is dispersed and placed on a glass slide. The particles are viewed through a microscope by means of transmitted light. The areas of the magnified images of the

*Rules for rounding off numerical values (*revised*).

†Specification for test sieves (*revised*).

‡The ISO series is 63, 45, 31, 22, 16, 11, 7.8, 5.5, 3.9, 2.8, 2.0, 1.4, 1.0. The optical microscope method as given here does not apply to this series. A different graticule would be needed.

§Glossary of terms relating to powders.

particles are compared with those of the reference circles of known size inscribed on a graticule and simultaneously visible. The relative numbers of particles in each of a series of size classes are determined. These numbers, expressed as percentages, constitute the size distribution by number. From the relative number in the size classes the relative volumes are calculated, assuming that particles of all sizes have the same shape. The relative volumes, expressed as percentages, constitute the size distribution by volume. The same is the size distribution by weight also if particles of all sizes have the same density.

3.2 Particle Shape—The great majority of particles may depart from the regular form to a greater or lesser extent the extremes being needles and plates. The measure of deviation which is called the shape factor is unimportant for particles of size below 75 microns except in the case of materials which are predominantly acicular in form. Such particles may be assessed according to their lengths and breadths in the manner (non-standard) as indicated in Appendix B.

3.3 The accuracy of size measurement is dependent upon the following:

- a) The optical quality of the images, which calls for the use of an adequate numerical aperture of the microscope objective and condenser for the size of the particle being measured;
- b) The accurate measurement of the magnification factor at the plane of the graticule on which the reference circles are inscribed;
- c) The reliability of comparison of the image areas with those of the reference circles;
- d) The uniformity of dispersion on the slide; and
- e) The number of particles counted.

4. SUB-DIVISION OF SAMPLE

4.1 Methods of preparing the laboratory sample so as to be representative of the gross sample are given in IS: 4879-1968*.

4.2 Method of Preparing the Analysis Sample—The sample for analysis is prepared as detailed in Appendix C.

4.3 Upper Limit of Size Measurement—This is determined by the increasing difficulty of measurement of the larger and larger particles, due to their thickness in relation to the depth of focus of the optical system, and by the availability of a suitable method of sieve analysis for such sizes. The optical microscope method is, therefore, preferably restricted to sizes of particles passing a 75-micron IS Sieve. Particles of size up to 150 microns may be counted and sized, provided that the weight of particles

*Methods of Sub-division of gross sample of powder used for determination of particle size.

of sizes retained on a 75-micron IS Sieve does not amount to more than 10 percent of the total weight of the powder. In case the weight exceeds 10 percent, these particles should be removed and analysed by sieving.

4.4 Lower Limit of Size Measurement—This is determined by the resolving power of the microscope. The method described here is applicable to particles of size 0.6 (more correctly 0.59) microns and above. Any smaller particles present in the sample should not be sized, but their presence should be stated in reporting the results.

4.5 Size Classes—Table 1 gives the recommended values of the size class limits which have been calculated from a base value of 53 microns.

5. APPARATUS

5.1 Microscope—The equipment shall be adequate for the work, the strictest specifications being imposed by the size of the smallest particles to be classified. The microscope used should be stable and be protected from vibration. All units of the microscope should be permanently attached to a common base. The numerical aperture should be adequate for the magnification involved.

5.1.1 The microscope (Fig. 1) should be provided with: (a) a source of illumination, (b) coarse and fine focusing, (c) focusing and centring substage condenser, (d) adjustable substage condenser diaphragm, (e) a mechanical stage with graduated movements at right angles, each capable of being read to 0.1 mm, (f) objectives to cover the whole ranges of sizes from 0.6 to 150 microns, (g) an eyepiece which should preferably be of focusing type, (h) a graticule and (j) a stage micrometer. The microscope to be used shall be of any of three following types:

- a) Bench microscope with a graticule mounted in the eyepiece. The microscope should be fitted with a graduated draw tube adjustable over a range preferably from 140 to 200 mm.
- b) Projection microscope with graticule mounted in the eyepiece. The microscope should be fitted with a graduated adjustable draw tube as for the bench microscope.
- c) Projection microscope with a graticule incorporated in or superimposed on a projection screen. The microscope may have either a graduated adjustable draw tube or an adjustable projection distance.

5.2 Microscope Lamp and Filters—The source of illumination used shall be a relatively homogenous light source and shall be capable of filling uniformly the whole field of view of the lowest power objective. It shall be provided with an adjustable diaphragm. Coloured and neutral filters are required for controlling colour and intensity of the illumination. The use of monochromatic illumination is recommended when sizing particles smaller than 2.3 microns.

TABLE 1 RECOMMENDED VALUES FOR SIZE CLASSES*
(Clauses 4.5, 5.6.2 and 7.3)

SIZE CLASS NUMBER	UPPER AND LOWER LIMITS TO SIZE CLASS IN MICRONS		ARITHMETIC MEAN OF SIZE CLASS LIMITS IN MICRONS (EXPRESSED TO THREE SIGNIFICANT FIGURES)	WEIGHTING FAC- TOR FOR CLASS (TO BE USED IN DERIVATION OF WEIGHT DISTRI- BUTION)†
	Expressed to Two Decimal Places	Rounded Figures		
(1)	(2)	(3)	<i>d</i> (4)	(<i>d</i>) ^a (5)
1	0.59 0.83	0.6 0.8 }	0.71	0.36
2	0.83 1.17	0.8 1.2 }	1.00	1.00
3	1.17 1.66	1.2 1.7 }	1.41	2.83
4	1.66 2.34	1.7 2.3 }	2.00	8.00
5	2.34 3.31	2.3 3.3 }	2.83	22.7
6	3.31 4.68	3.3 4.7 }	4.00	64.0
7	4.68 6.63	4.7 6.6 }	5.66	181
8	6.63 9.37	6.6 9.4 }	8.00	512
9	9.37 13.25	9.4 13.0 }	11.3	1 450
10	13.25 18.74	13 19 }	16.0	4 100
11	18.74 26.50	19 27 }	22.6	11 600
12	26.50 37.48	27 37 }	32.0	32 800
13	37.48 53.00	37 53 }	45.2	92 700
14	53.00 74.95	53 75 }	64.0	262 000
15	74.95 106.00	75 106 }	90.5	741 000
16	106.00 149.90	106 150 }	128	2 100 000

*Recommended values constitute a $\sqrt{2}$ series based on 53.00 microns (see 4.4).

†The values for weighting factor *d*^a are computed from the means of the class limits (second column), not from the rounded means given in the fourth column.

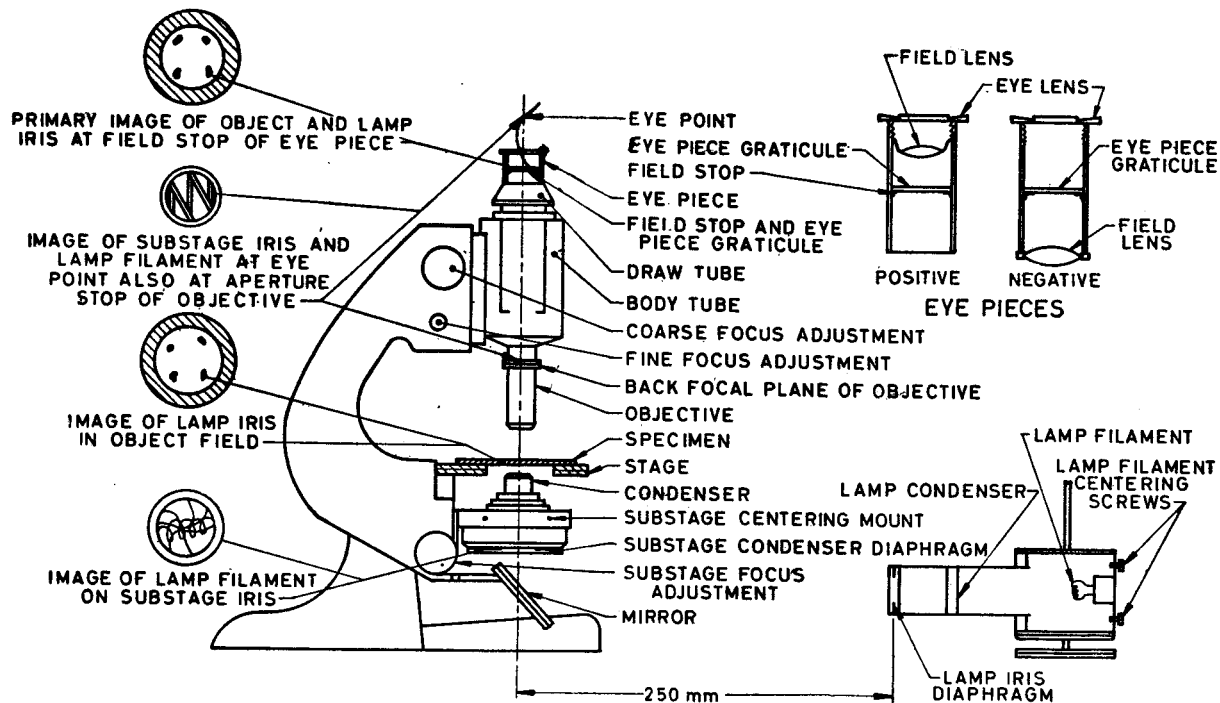


FIG. 1 MICROSCOPE AND LAMP

5.3 Objectives — The objectives shall be of good quality, in sound condition and inscribed with their focal length and numerical aperture. Objectives may be achromatic, fluorite or apochromatic. It may be necessary to have available at least 3 objectives to cover the whole range of sizes from 0.6 to 150 microns.

5.4 Eyepiece — Only one eyepiece is required. It shall be of good quality, in sound condition and inscribed with its magnification. Eyepieces may be of Huygenian (negative) or compensating (negative or positive) type. Compensating eyepieces should be employed with apochromatic fluorite and the higher power achromatic objectives. Eyepieces in which graticules are mounted should preferably be of positive construction. The lens should be adjustable within its mount to permit the focusing of the graticule when resting on the field stop. The eyepiece of a bench microscope should preferably be of power $\times 20$ but not greater than $\times 25$.

5.5 Substage Condenser — The substage condenser shall be of good quality, in sound condition and shall have numerical aperture at least as large as the numerical aperture of the objective in use. It may be necessary to have available more than one substage condenser. As it may not be possible to illuminate the whole field of view of low power objectives by means of condenser having a numerical aperture adequate for high power objectives, a three-lens aplanatic condenser of 1.3 numerical aperture corrected for spherical and chromatic aberrations, whose numerical aperture may be reduced by removing the top lens, is suitable for all objectives.

5.6 Graticule — The graticule (Fig. 2) shall be ruled with rectangles and reference circles to the pattern and relative dimensions specified in IS:5257-1969*.

5.6.1 The eyepiece graticule shall be of such diameter as to fit without excessive play into the eyepiece used. A screen graticule shall be of such size as to be contained within the field of view at the projection distance employed; the field of view will usually have a diameter of about 150 mm at a projection distance of 250 mm measured from the eyepiece and will be proportionally larger for longer distances.

5.6.2 The diameters of the reference circles should preferably be such that, within the available range of adjustment of magnification which is achieved by tube length movement for eyepiece graticules or projection distance for screen graticules, the diameters shall correspond at the field with the dimensions stated in col 2 of Table 1 (see 1.1).

5.7 Stage Micrometer — The stage micrometer shall have 100 divisions each of 10 microns.

5.8 Procedure for Adjustment of Microscope — The recommended procedure for adjustment of microscope is given in Appendix D.

*Specification for eyepiece and screen graticules for the determination of the particle size of powders.

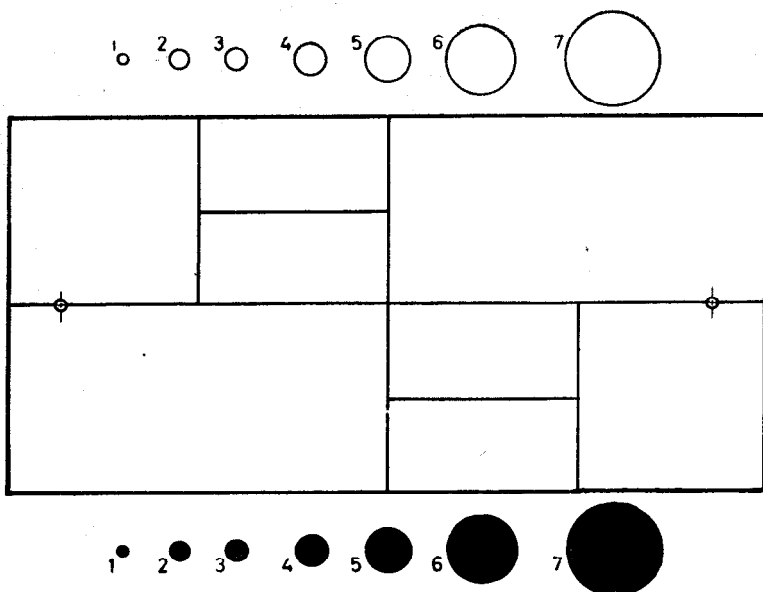


FIG. 2 INDIAN STANDARD GRATICULE

6. SELECTION OF OBJECTIVES AND EYEPIECES

6.1 Minimum Numerical Aperture — An objective shall not be employed for sizing of particles below the minimum particle size considered appropriate to its nominal numerical aperture as given in Table 2. The minimum particle size in microns is equal to 1.5 divided by the nominal numerical aperture (except in the case of an apochromatic objective of numerical aperture 1.3 and above). This condition ensures that the diameter of the smallest particle examined will be some five times that of the airy disc. It is recommended that wherever possible sizing should not be extended below the size class given in col 6 of Table 2.

6.1.1 The manufacturer's rated value for the numerical aperture as inscribed in the objective may be taken for the numerical aperture. When the rated value exceeds unity the particles should be mounted in a medium of refractive index greater than 1.3 and both condenser and objective should be oiled to the slide by immersion oil. If, on the other hand, the particles are dry mounted, or the condenser or objective is not oiled to the slide, the nominal numerical aperture shall be taken as 1.0 or as the rated value whichever is smaller. The fact that the condenser will be stopped down in use so that the actual or working numerical aperture is less than its nominal value does not reduce the minimum particle size which may be measured with the objective.

**TABLE 2 MINIMUM PARTICLE SIZE FOR DIFFERENT OBJECTIVES AND
MINIMUM TOTAL MAGNIFICATION REQUIRED**

(Clause 6.1)

FOCAL LENGTH OF OBJECTIVE	TYPE OF OBJECTIVE*	NOMINAL MAGNIFICA- TION OF OBJECTIVE	NOMINAL NUMERICAL APERTURE, NOT LESS THAN	MINIMUM PERMISSIBLE PARTICLE SIZE MICRONS	RECOMMEND- ED SMALLEST SIZE CLASS MICRONS	MINIMUM TOTAL MAGNIFI- CATION REQUIRED FOR SIZING DOWN TO	
						Minimum Particle Size	Recommend- ed Smallest Size Class
mm							
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
<i>Dry Objectives</i>							
32	Achromatic	× 3.5	0.10	15	19 - 27	100	80
	Achromatic	× 5	0.13	12	19 - 27	130	80
25	Achromatic	× 6.5	0.15	10	19 - 27	150	80
16	Achromatic	× 10	0.17	8.8	13 - 19	170	115
	Apochromatic	× 10	0.30	5.0	9.4 - 13	300	160
12	Achromatic	× 12.5	0.34	4.4	6.6 - 9.4	340	230
8	Achromatic	× 20	0.50	3.0	4.7 - 6.6	500	320
	Apochromatic	× 20	0.65	2.3	3.3 - 4.7	650	450
6	Achromatic	× 25	0.50	3.0	4.7 - 6.6	500	320
4	Achromatic	× 40	0.65	2.3	3.3 - 4.7	650	450
	Apochromatic	× 40	0.85	1.8	2.3 - 3.3	850	650
<i>Oil Immersion Objectives</i>							
3.75 } 3.6 }	Fluorite	× 45	0.95	1.6	2.3 - 3.3	950	650
2.2 } 2.0 }	Achromatic	× 80 - 100	1.25	1.2*	1.7 - 2.3†	1 250	880
1.8 }	Apochromatic		1.30	0.6*	0.8 - 1.2†	2 500	1 800

*Achromatic includes parachromatic; apochromatic includes fluoride and semi-apochromatic.

†Providing particles are mounted in a medium of refractive index greater than 1.3 and both condenser and objective are oiled. Otherwise, in both cases, the minimum particle size is increased to 1.5 microns and the recommended smallest size class to 2.3 - 3.3 microns, the minimum total magnifications become 1 000 and 650.

6.2 Minimum Total Magnification of Objective and Eyepiece—The total magnification of the objective and eyepiece together shall be such, that the magnified image of the smallest particle being sized exceeds 1.5 mm. This condition ensures that the magnified image is not less than about 20 times the limit of resolution of the unaided eye.

6.3 Maximum Total Magnification of Objective and Eyepiece—The total magnification employed shall not exceed 1 500 times the numerical aperture of the objective. There is no advantage to be gained by further increasing the magnification, as no further detail in the object may usually be made visible to the eye.

6.4 Choice of Objectives

6.4.1 Number Size Distribution—For sizing down to 0.6 microns a convenient series of objectives is given in Table 3.

TABLE 3 RECOMMENDED SERIES OF OBJECTIVES FOR PARTICLES IN THE SIZE RANGE FROM 0.6 TO 150 MICRONS

PARTICLE SIZE RANGE IN MICRONS	FOCAL LENGTH OF OBJECTIVE
0.6 to 4.7	2 mm apochromat (oil immersion)
4.7 to 27	8 mm achromat (or apochromat)
27 to 150	25 mm (or 32 mm) achromat

6.4.2 If it is not necessary to size below 2.3 microns the use of oil immersion may be dispensed with and a 4 mm objective can be used as of the highest power. The series then will be as given in Table 4.

TABLE 4 RECOMMENDED SERIES OF OBJECTIVES FOR PARTICLES IN THE SIZE RANGE FROM 2.3 TO 150 MICRONS

PARTICLE SIZE RANGE IN MICRONS	FOCAL LENGTH OF OBJECTIVE
2.3 to 13	4 mm apochromat (or achromat)
13 to 37	16 mm achromat (or apochromat)
37 to 150	25 mm (or 32 mm achromat)

6.4.3 Weight Size Distribution—Particles smaller than 2.3 micron often do not contribute significantly to weight size distribution and in such cases the series of objectives as given in Table 4 may be used. However, if the weight of particle smaller than 2.3 microns is of importance the use 2 mm oil immersion objective becomes necessary.

7. SELECTION OF GRATICULE AND ADJUSTMENT OF MAGNIFICATION

7.1 Standard Graticule—A standard graticule (*see* Fig. 2) ruled with a rectangular grid shall be used to define the area of the field of view within which particles are counted. The graticule is provided with two sets of numbered circles ranging from $\sqrt{2}$ to $8\sqrt{2}$ units in diameter, which serve as reference circles for classifying by size the particles counted.

7.1.1 The relative dimensions of the grid and the circles shall be as given in Table 5. The diameters of the reference circle increase in geometrical progression with a constant ratio of $\sqrt{2}$. The physical size of graticule rulings is designated by the length of the rectangular grid which is $\frac{100}{\sqrt{2}}$ times the diameter of the circle numbered 1. This length is marked on the graticule in millimeters to an accuracy of ± 2 percent.

TABLE 5 RELATIVE DIMENSIONS OF STANDARD GRATICULE

	NUMERICAL VALUE UNITS*
Grid length	100
Grid breadth	50
Distance between marks	85.4
Diameter of circle	
1	1.41
2	2.00
3	2.83
4	4.00
5	5.66
6	8.00
7	11.31

*The unit is 1/100th of the grid length.

7.1.2 The grid is also sub-divided to define smaller fields of view two each 1/4 and 1/8 and four each 1/16 of the total area of the grid for use when required. The graticule is also provided with two calibration marks at a specified distance apart on the longer axis of the grid for adjusting the magnification of the microscope (*see* IS:5257-1969*).

7.2 Choice of Grid Length of Eyepiece Graticule—The procedure for selecting the length of grid of an eyepiece graticule is described in Appendix E.

*Specification for eyepiece and screen gratiules for the determination of the particle size of powders.

7.3 Matching of Reference Circle with Size Class Limits—The magnification of the microscope shall be so adjusted that the diameters of the reference circles correspond to the desired size class limits as given in Table 1. The series of possible matchings of the reference circles with the recommended size class limits in the range from 0.6 to 150 microns is given in Table 6. At any one matching up to 7 size classes covering a range of $8\sqrt{2}$ to $\sqrt{2}$ may be counted and classified. At least 3 different matchings are needed to cover the whole size range from 0.6 to 150 microns.

7.3.1 The matching is carried out by adjusting the magnification at the plane of the graticule in relation to the physical size of the reference circles as characterized by the length of the grid inscribed on the graticule. Since it is desirable, as a matter of manipulative convenience, to employ a single graticule having a length of grid suitable for the various matchings required in any particular case, the magnification at the plane of the graticule should be adjustable in appropriate steps which are powers of $\sqrt{2}$. The relative magnifications required for this purpose are given in col 1 of Table 6. The matching of the largest set of size classes, which has been assigned a relative magnification of unity, is usually made with a 32 mm or a 25 mm objective having an actual magnification of about $\times 4$ to $\times 6$. Any other objective used for the other size classes shall be assigned the relevant relative magnification in the $\sqrt{2}$ series (col 1 of Table 6) depending upon its approximate magnification at eyepiece graticule obtained at 160 mm tube length. The nominal magnification which is inscribed on the objective by the manufacturer may be judiciously made use of for this purpose. In effecting the matchings, account should always be taken of the conditions governing the use of the various objectives (6.1 and Table 2). Column 12 in Table 6 gives the minimum numerical aperture of objective which may be used for matching and the last column gives the minimum total magnification.

7.4 Choice of Grid Length of Screen Graticule and Adjustment of Magnification for Microscope With Screen Graticule—The choice of the length of grid to suit the different matchings for covering the whole size range presents no difficulty in the case of a screen graticule. There is, however, a minimum length of grid imposed by the condition (6.2) that the smallest circle (circle 1) shall have a diameter of not less

than 1.5 mm. As the length of the grid is $\frac{100}{\sqrt{2}}$ times the diameter of circle 1, the graticule must be of such size that the length of the grid is not less than 106.1 mm.

7.4.1 Procedure for Adjusting the Magnification to Obtain a Matching of the Reference Circles with the Recommended Size Classes—Select an objective having

numerical aperture larger than the minimum permitted for the matching and an eyepiece sufficient to give, with the selected objective a total magnification larger than the minimum shown in Table 6. Place the stage micrometer (*see* 5.7) on the microscope stage and align it with the longer axis of the grid on the screen graticule. Adjust the magnification of the microscope by altering the projection distance until there is an exact correspondence between the distance separating the two calibration marks on the graticule and the length on the stage micrometer specified in Table 6 for the matching. Repeat the process whenever the objective or eyepiece is changed. The optical conditions when adjusting the magnification including the colour of the illumination and the refractive index of the immersion oil, if used, shall remain the same as those when the slide of particles replaces the stage micrometer.

7.5 Choice of Grid Length of Eyepiece Graticule and Adjustment of Magnification for Microscope with Eyepiece Graticule—

The choice of length of grid to suit the several different matchings required for covering the whole size range is governed by the characteristics of the objectives, eyepiece and microscope available. The magnification at the graticule is determined by the magnification of the objective (and also on the power of the field lens in the case of a negative eyepiece) at a given tube length. The magnification of objectives of different focal lengths, as inscribed on the objectives, do not correspond sufficiently closely with terms in the required $\sqrt{2}$ progression for use at the same tube length. It is, however, usually possible to bring them into the exact correspondence required as in Table 6 by adjusting the tube length of the microscope provided it has a sufficient range of movement. There will be often little scope for tube length adjustment to secure matching with the recommended size class unless the length of grid has been suitably chosen. The method of effecting the choice of suitable grid length is described in Appendix E. The grid length should not be more than five-eighths the diameter of the field of view (diameter of the field stop of the eyepiece). If a graticule having a suitable grid length is not available, there is no alternative but to match the reference circles with size classes other than those recommended in Table 6.

7.5.1 Procedure for Adjusting the Magnification to Obtain Matching of the Reference Circle with the Recommended Size Classes—Select an objective of the required focal length for the matching desired from Table 7. Select an eyepiece of appropriate power to comply with the conditions as given in 6.2 and 6.3 and Table 2. The minimum eyepiece power required with a bench microscope to magnify the various circles up to the specified minimum size of 1.5 mm are shown at the foot of Table 7. Select a graticule of appropriate length as detailed in Appendix E and insert it in the eyepiece above the field stop and focus it by moving the eyepiece lens system (eye lens only in the case of negative eyepiece) relative to the graticule.

**TABLE 6 COMPLETE SET OF MATCHINGS OF REFERENCE CIRCLES
WITH SIZE CLASS LIMITS**

[*Clauses 7.3.1, 7.4, 7.5 and D-1.2 (h)*]

RELATIVE MAGNIFI- CATION AT PLANE OF STANDARD GRATICULE	SIZE CLASS LIMIT IN MICRONS CORRESPONDING TO GRATICULE REFERENCE CIRCLE NUMBERED								LENGTH OF STAGE MIC- ROMETER TO BE MATCHED WITH CALI- BRATION MARKS ON GRATICULE MICRONS	AREA OF RECTANGU- LAR GRID OF GRATICULE sq. mm	MINIMUM NOMINAL NUMERICAL APERTURE OF OBJECTIVE	MINIMUM TOTAL MAG- NIFICATION
	*	7	6	5	4	3	2	1				
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
1†	150	106	75	53	37	27	19	13	800	0.439 0	0.11	115
$\sqrt{2}$	106	75	53	37	27	19	13	9.4	566	0.219 5	0.16	160
2	75	53	37	27	19	13	9.4	6.6	400	0.109 8	0.23	230
$2\sqrt{2}$	53	37	27	19	13	9.4	6.6	4.7	283	0.054 88	0.32	320
4	37	27	19	13	9.4	6.6	4.7	3.3	200	0.027 44	0.45	450
$4\sqrt{2}$	27	19	13	9.4	6.6	4.7	3.3	2.3	141	0.013 72	0.65	650
8	19	13	9.4	6.6	4.7	3.3	2.3	1.7	100	0.006 859	0.90	900
$8\sqrt{2}$	13	9.4	6.6	4.7	3.3	2.3	1.7	1.2	71	0.003 430	1.25	1 250
16	9.4	6.6	4.7	3.3	2.3	1.7	1.2	0.8	50	0.001 715	1.30	1 800
$16\sqrt{2}$	6.6	4.7	3.3	2.3	1.7	1.2	0.8	0.6	35	0.000 857 4	1.30	2 500

*The standard graticule is not inscribed with a reference circle of diameter corresponding to the size class limits in col 2, which are $\sqrt{2}$ times larger than those listed under reference circle 7, but particles may be judged as being larger than reference circle 7 whilst smaller than an imaginary circle $\sqrt{2}$ times larger.

†This matching is made with a 32 mm or a 25 mm objective which usually has an actual magnification of $\times 4$ to $\times 6$.

TABLE 7 SELECTED SET OF MATCHINGS OF REFERENCE CIRCLES WITH SIZE CLASS LIMITS (FOR USE WITH MICROSCOPES HAVING EYEPIECE GRATICULES) AND MINIMUM EYEPIECE POWER FOR BENCH MICROSCOPE

(Clauses 7.5.1, 7.5.2 and E-1.1)

FOCAL LENGTH	NOMINAL NUMERICAL APERTURE NOT LESS THAN		APPROXIMATE MAGNIFICATION AT EYEPIECE GRATICULE OBTAINED AT 160 mm TUBE LENGTH	RELATIVE MAGNIFICATION AT EYEPIECE GRATICULE REQUIRED FOR MATCHING	SIZE CLASS LIMIT IN MICRONS CORRESPONDING TO GRATICULE REFERENCE CIRCLE NUMBERED								LENGTH ON STAGE MICROMETER REQUIRED FOR MATCHING WITH CALIBRATION MARKS ON EYEPIECE GRATICULE
	For Achromatic Objectives	For Apochromatic Objectives			*	7	6	5	4	3	2	1	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
<i>Dry Objectives</i>													
mm													Microns
32 $\frac{1}{2}$	0.10 } 0.15 }	—	4	1	150	106	75	53	37	27	19	13†	800
25		—	5 or 6										
16	0.17	0.30	9 or 10	2	75	53	37	27	19	13	9.4†	6.6†	400
8	0.50	0.65	20	4	37	27	19	13	9.4	6.6	4.7	3.3†	200
4	0.65	0.85	40	8	19	13	9.4	6.6	4.7	3.3	2.3†	1.7†	100
<i>Oil Immersion Objectives</i>													
3.75 } or 3.6 }	—	0.95	40, 45 or 50	8	19	13	9.4	6.6	4.7	3.3	2.3†	1.7†	100
2.2, } 2.0 } or 1.8 }	1.25	1.30	80, 95 or 100	16 $\sqrt{2}$	9.4	6.6	4.7	3.3	2.3	1.7	1.2†	0.6†	35

Minimum Eyepiece Power for Bench Microscope

Minimum eyepiece power required to magnify the various circles up to the specified minimum size of 1.5 mm when for unit relative magnification, the approximate magnification at eyepiece graticule is:	4 (32 mm)	7×	10×	15×	20×	25×
	5 (25 mm)	6×	8×	12×	16×	23×
	6 (25 mm)	5×	7×	10×	14×	19×

*The standard eyepiece graticule is not inscribed with a reference circle of diameter corresponding to the size class limits in col 6 which are $\sqrt{2}$ times larger than those listed under reference circle number 7, but particles may be judged as being larger than reference circle 7 whilst smaller than an imaginary circle $\sqrt{2}$ times larger.

†Not recommended although permissible for achromatic objectives (as defined in foot-note in Table 2).

‡Not recommended although permissible for achromatic objectives (as defined in foot-note in Table 2); not permissible for achromatic objectives.

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7.5.2 Place the stage micrometer (*see* 5.7) on the microscope stage and align it with the longer axis of the grid on the graticule. The stage micrometer should be focussed AFTER focussing the eyepiece graticule for bench microscopes and BEFORE for projection microscopes. Adjust the magnification of the microscope by altering the tube length until there is exact correspondence between the distance separating the two calibration marks on the graticule and the length on the stage micrometer as specified in Table 7. Repeat the process whenever the objective or eyepiece is changed. The optical conditions when adjusting the magnification, including the colour of the illumination and refractive index of the immersion oil, if used, shall be the same as those obtaining when the slide of particles replaces the stage micrometer.

* **7.5.3** If it is not possible to effect the adjustment within the range of tube length movement or if the reference circles are to be matched with size class limits other than those given in Table 6, the magnification at the graticule shall be measured by means of the stage micrometer and the corresponding size classes calculated. For this purpose the length of the grid, or the distance between the calibration marks, may be compared with the divisions of the stage micrometer. High power objectives should be used as far as possible near the tube length for which they have been corrected in order to obtain optimum optical performance.

8. DETERMINATION OF NUMBER OR WEIGHT SIZE DISTRIBUTION

8.1 Preparation of the analysis Sample—The sample is prepared in the manner as indicated in 4.2. The concentration of particles on the slide should be so adjusted when preparing it that each field of view contains on an average about six particles of the size class being examined. In general, a lower concentration is required when determining the number size distribution than when determining the weight size distribution.

8.2 Adjustment—The microscope should be set up and adjusted in the manner as detailed in Appendix D. It is desirable to employ monochromatic illumination if particles of size smaller than 2 microns are to be included in the measurements. The appropriate objectives, eyepiece and graticule should be selected as stated under 5, 6 and 7. The magnification should be adjusted by the appropriate method given in 7.4 and 7.5. The slide with particles should be mounted on the mechanical stage of the microscope. The field of view should be adjusted by means of lamp iris to an area not much larger than that occupied by the grid and the reference circles.

8.3 Counting Procedure—As it is not practicable to count and classify every particle on the slide, only a sample out of the total number is examined at any time. The sample is contained in a number of field

areas defined by the boundaries of the graticule grid or a sub area of it as determined by the conditions specified in 13 and 14 which are designed to take account of the errors of sampling and also to avoid counting more particles than necessary for securing an acceptable degree of precision. The conditions are different in the determination of the number and of the weight size distribution.

8.3.1 As the concentration of the particles per unit area may vary from place to place even when the slide is prepared most carefully, the field areas in which the particles present are to be counted should always be spaced out in a regular pattern in such a way that each represents an equal proportion of the total area of the slide covered by particles. The pattern used will depend on the number of fields to be counted. The procedure is illustrated in Fig. 3 which shows how 25 sample fields are considered in an area of 20 mm \times 20 mm on the slide.

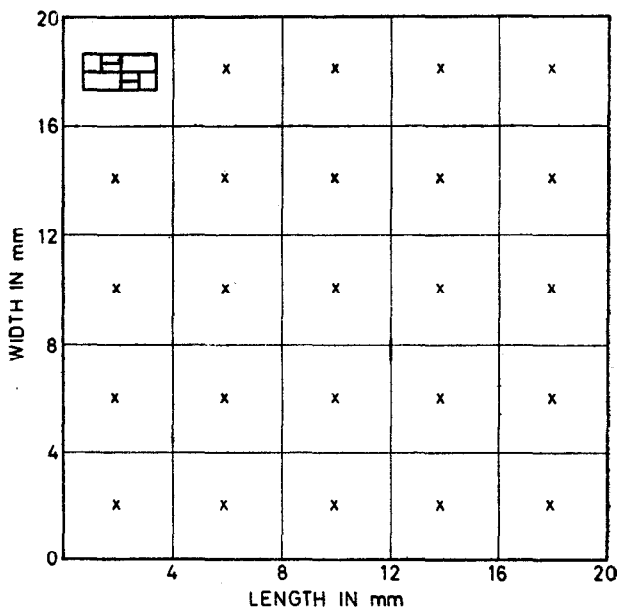
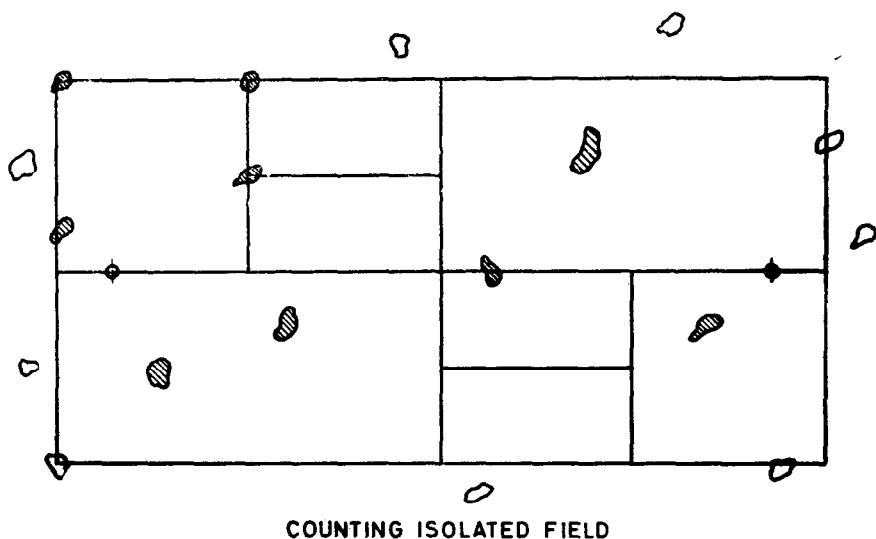


FIG. 3 LOCATION OF SAMPLE FIELD

8.3.2 The basic technique of measurement by comparison with the reference circles on the graticule is to match them by mentally squeezing each particle into the circle of appropriate diameter. Every particle present in each field area is examined individually, the focus being adjusted if necessary, and its area compared mentally with the area of the reference circles on the graticule. The matching process should thus be carried out without moving the particles from the positions which they

occupy within the rectangle of the graticule. A particle whose area is estimated to be smaller than that of, say, circle 5 but larger than that of circle 4 is assigned to the size class defined by the diameter of the circles 4 and 5 and so on for other size classes. The transparent particles are compared with the open circles and opaque particles are compared with the solid circles. In order to avoid counting the same particle twice when sizing in adjacent areas, all the particles are recorded as belonging to the field area if they lie wholly within the boundary lines of the field area and also if they are in contact, however, slightly, with the top and left hand edges of the rectangle, similarly ignoring all those touching the bottom and right hand edges as shown in Fig. 4. It is recommended that, as far as is practicable, the particles of all the size classes being examined at one magnification should be assigned to their respective classes before passing on to the next field area.



Shaded particles are included in the count. Unshaded particles are excluded from count.

FIG. 4 TREATMENT OF EDGE PARTICLES

8.3.3 If particles of the sizes being considered occur only rarely, it is recommended that the sample be taken in the form of long narrow strips each 5-20 mm long and of width defined by the upper and lower edges of the graticule grid. This technique will be found specially useful for counting the largest 3 or 4 size classes in the determination of weight size distribution.

9. CALCULATIONS

9.1 Size Distribution—The size distribution shall be calculated from the number of particles m_r counted in the size classes of mean size d_r found in the sample areas $n_r a_r$,

where

n_r = number of fields examined, and

a_r = area of each field in mm^2 .

9.2 Number Size Distribution—The number of particles per unit area, $m_r/n_r a_r$ for each size class shall be calculated and the sum $\Sigma \frac{m_r}{n_r a_r}$ for all size classes obtained. The percentage by number in each size class is given by:

$$P_r = \frac{100 (m_r / n_r a_r)}{\Sigma m_r / n_r a_r}$$

9.2.1 The number percentage over or under each size class limit is obtained by summing the percentage numbers in all larger or smaller size classes as illustrated in the example given in Appendix F.

9.3 Weight Size Distribution—The number of particles per unit area $\frac{m_r}{n_r a_r}$ for each size class shall be calculated and multiplied by the cube (d_r^3) of the mean size. The sum of the products for all the size classes,

$$\Sigma \frac{m_r d_r^3}{n_r a_r} \text{ is obtained.}$$

Their percentage by weight in each size class is given by:

$$Q_r = \frac{100 (m_r d_r^3 / n_r a_r)}{\Sigma m_r d_r^3 / n_r a_r}$$

9.3.1 The weight percentage over or under each size class limit is found by summing the percentage weights in all larger or smaller size classes as illustrated in the example given in Appendix G.

10. STANDARD ERROR

10.1 The expected standard error is a measure of the errors due to counting and classifying only a limited number of particles out of the total number present on the slide sample. It should be kept less than 2 percent in order that the reproducibility or the determination on different slides may be within the given limits specified in 11.

10.2 Number Size Distribution—The expected standard error $S(P_r)$ of the percentage P_r by number in each size class, out of the total number in all size classes is:

$$S(P_r) = \sqrt{\frac{P_r(100 - P_r)}{\Sigma m_r}}$$

The standard error is maximum when $P_r = 50$ and, therefore, it will always be less than 2 percent if the total number of particles of all sizes counted, $\Sigma(m_r)$ is greater than 625.

10.3 Weight Size Distribution—The expected standard error $S(Q_r)$ of the percentage Q_r by weight in each size class, out of the total weight in all size classes, is given approximately by:

$$S(Q_r) = \frac{Q_r}{\sqrt{m_r}} \sqrt{1 - \frac{Q_r}{50}}$$

The expected standard error calculated with the help of the above formula for each size class should not exceed 2 percent.

11. REPRODUCIBILITY

11.1 The size distribution shall be measured on not less than two analysis samples. The percentage of material, in each size class, by number or by weight according to the type of size distribution desired shall be calculated in the manner as stated in 9.2 and 9.3. For each class the range of these percentages shall be calculated and from these the arithmetic average of these ranges for all size classes shall be obtained. The range of the percentages for each individual class so calculated shall not be greater than the limit given in Table 8 Col 2 for the specified number of analysis samples counted. If the range for all size classes and the average range are within the limits set out in Table 8 the arithmetic averages of the percentages calculated for the individual analysis samples shall be taken as the final size distribution. In case the range for any size class or the average range for all size classes exceeds the corresponding limit given in Table 8, then not less than two further analysis samples shall be counted. In the unlikely event of having to examine more than seven analysis samples the results shall be classified into two or more groups according to the order in which the counts have been made. No such group shall consist of the results of less than 4 analysis samples. The limits set out in Table 8 should be applied to each group separately.

12. REPORTING RESULTS

12.1 Results shall be reported as percentage oversize or percentage under size, the percentage being given to the nearest one percent. It shall be clearly stated whether the results reported refer to number size distribution or weight size distribution. The limits of size classes shall be given in microns to two significant figures. The criteria adopted in classifying aggregates or their constituent particles shall be stated. The presence of

appreciable number of particles of size less than 0.6 microns, if any, should be stated in reporting the results. As far as weight size distribution is concerned the presence of particles of size below 0.6 microns is of no consequence.

TABLE 8 RANGES FOR SIZE CLASSES

(Clause 11.1)

NUMBER OF ANALYSIS SAMPLES COUNTED	MAXIMUM VALUE FOR RANGE OF PERCENT- AGE IN ANY SIZE CLASS	MAXIMUM VALUE FOR AVERAGE RANGE OF PERCENTAGES FOR ALL SIZE CLASSES		
		Not More than 4 Size Classes	5 to 8 Size Classes	9 or More Size Classes
(1)	(2)	(3)	(4)	(5)
2	6.0	4.5	4.0	3.7
3	7.5	6.0	5.5	5.2
4	9.0	7.5	6.8	6.3
5	9.5	8.0	7.4	7.0
6	10.0	8.5	7.9	7.5
7	10.5	9.0	8.2	7.8

13. CONDITIONS GOVERNING THE DETERMINATION OF THE NUMBER SIZE DISTRIBUTION

13.1 The conditions to be satisfied in determining the number size distribution are:

a) the standard error (as stated in **10**) of the percentage by number in each size class shall be less than 2 percent of the total number in all size classes, for each of the analysis samples examined; and

b) the reproducibility of determination on two or more analysis samples shall satisfy the requirements as specified in **11**.

The requirements in **13.1.2** and **13.1.3** ensure that condition (a) will be satisfied and requirements in **13.1.1**, **13.1.4**, **13.1.5**, **13.1.6** and **13.1.7** are designed to ensure that condition (b) is also met with.

13.1.1 Control Size Class—The size class that contains the highest percentage by number of particles shall be considered as control size class. This may usually be the smallest size class. If the most frequent size class is not known and could not be deduced from a preliminary inspection of the slide, the smallest size class present in the slide sample shall be considered as the control size class. The control size class is denoted by the subscript 'o', the mean size of the class by d_o , the number of particles counted and classified as belonging to it by m_o and the sample area from which they are drawn by $n_o a_o$.

13.1.2 Minimum total Number of Particles To be Counted—The total number of particle of all size classes counted and classified shall be not less than 625.

13.1.3 Sample Area—The sample area (the product $n_r a_r$ of the number n_r of fields each of are a_r) examined for particles of each size class shall be constant.

13.1.4 Minimum Number of Fields—The number of fields n_o examined for particles of the control size class shall not be less than 96. The number of fields examined for particles of any other size class shall not be less than 12 and preferably not less than 24.

13.1.5 Area of the Field—The area a_o of each field examined for particles of the control size class shall be that corresponding to the whole rectangular grid of the graticule. This condition applies also for all size classes examined at the magnification used for the control size class. The area a_r of each field examined for particles of other size classes at a different magnification shall be so chosen that the sample area $n_r a_r$ remains the same as that for the control size class $n_o a_o$ and also that the number n_r of fields exceeds 12.

13.1.6 Density of Fields—The concentration of particles on the slide should be so adjusted when preparing it that each field of area a_r contains on an average not more than about six particles of the size class being examined. It is desirable to adjust the concentration to give about three particles per field in the control size class. If the concentration is higher it is likely, for the number and area of fields specified above, that the total number of particles counted will unnecessarily exceed the minimum of 625.

13.1.7 Order of Examining the Size Classes—It is recommended that the size classes be examined in turn starting with the control size class, which for number size distribution will usually be the smallest size class. The other size classes to be examined at that magnification should be counted and classified at the same time over the same field area and the same number of fields. The magnification should then be reduced for examining the next larger size classes.

14. CONDITIONS GOVERNING THE DETERMINATION OF THE WEIGHT SIZE DISTRIBUTION

14.1 The conditions to be satisfied in determining the weight size distribution are:

- a) the standard error as stated in 10 of the percentage by weight in each size class shall be less than two percent of the total weight in all size classes, for each analysis sample examined; and

- b) the reproducibility of measurements on two or more analysis samples shall satisfy requirements as specified in 11. The requirements given in 14.1.1 to 14.1.8 are designed to ensure that in most cases both these conditions are satisfied.

14.1.1 Control Size Class — The largest size class which contains more than 5 percent by weight of particles shall be considered as control size class. If the largest size class containing more than 5 percent by weight is not known and cannot be identified from a preliminary inspection of the slide, the largest size class present in the slide sample shall be considered as control size class.

The control size class is denoted by subscript '0', the mean size of the class by d_0 , the number of particles counted and classified as belonging to it by m_0 and the sample area from which they are drawn by $n_0 a_0$, as in the case of number size distribution.

14.1.2 Minimum Number of Particles Counted in Control Size Class — The number of the particles counted and classified, m_0 as belonging to control size class shall be not less than 25. In case the control size class contains more than 10 percent by weight, which is generally unlikely, the number of particles to be counted in the control size class should exceed 25. The required number of particles ' m_r ' to be counted for the standard error to be less than 2 percent is given by equation given in 10.3.

14.1.3 Sample Area for the Control Size Class — The sample area $n_0 a_0$ to be examined for particles of the control size class may be the whole area of the slide occupied by the particles or may even require the use of more than one slide. In the latter case it is essential that the two slides are prepared from the same analysis sample so as to be of the same density.

14.1.4 Sample Area for Other Size Classes — The sample area $n_r a_r$ which is examined for particles of size classes other than the control size class shall be equal to or greater than:

$$n_0 a_0 \left(\frac{d_r}{d_0} \right)^6 \frac{N_r}{N_0}$$

where $n_0 a_0$ is the sample area examined for the control size class. The number concentration N_0 in the control size class is $\left(\frac{m_0}{n_0 a_0} \right)$ and the number concentration N_r in the r th class is $\left(\frac{m_r}{n_r a_r} \right)$. It is, therefore, necessary during the counting of each size class to estimate the concentration N_r which will be found when counting of the class has been completed (see the worked out example in Appendix G).

An alternative way of expressing the condition is that the factor

$$F_r = d_r^3 \sqrt{\frac{m_r}{n_r a_r}} \text{ should be smaller than the corresponding factor}$$

$$F_o = \frac{d_o^3 \sqrt{m_o}}{n_o a_o} \text{ for the control size class. The factor } F \text{ is}$$

referred to as the accuracy factor and is conveniently expressed in the form

$$F_r = \frac{N_r d_r^3}{\sqrt{m_r}}$$

14.1.5 Minimum Number of Fields—The number of fields n_o examined for particles of the control size class shall be not less than 96. It is recommended that the same minimum number of fields be examined for all size classes counted and classified at the magnification used for the control size class. The number n_r of fields examined for particles of any size shall be not less than 12 and preferably not less than 24.

14.1.6 Area of Each Field—The area of each field examined for particles of the control size class shall be that of corresponding to the whole rectangular grid of the graticule. The area of each field examined for other size class shall be adjusted according to the rules given in 14.1.4 and 14.1.5 and as illustrated by the example in Appendix G.

14.1.7 Density of Field—The concentration of particles on the slide shall be so adjusted when preparing it that each field contains, on the average, not more than about six particles of the size class being examined.

14.1.8 Order of Examining the Size Classes—It is recommended that the size classes be examined in turn starting with the control size class which for weight size distribution will usually be the largest size class. Also, it is recommended that not more than the next three smaller size classes be examined at the same magnification. The magnification should then be increased for examining the next smaller size classes.

APPENDIX A

(Clause 0.3)

CORRELATION OF RESULTS FROM DIFFERENT METHODS OF SIZE DETERMINATION

A-1. GENERAL

A-1.1 A number of methods are described for the determination of particles size in the range 1-1 000 microns. These are the microscope, elutriation, sedimentation and test sieving (IS:460-1962*). As no single method is

*Specification for test sieves (revised).

applicable to the whole range of sizes 1-1000 microns, it is necessary to combine the analysis by two or more different methods in order to establish the distribution in full. In general a particle size determination will consist of a sieving of the material down to 75 microns combined with an analysis of the fraction below that size by one of the other three methods. In the size determination, what is measured is not the particle size directly but only some size-dependent property of the particle. In the microscope method it is the enlarged image of the particle, in sieving it is the ability of the particle to pass through an aperture of given size, in sedimentation it is the fall speed through a stationary fluid and in elutriation it is the upward velocity of a fluid necessary to prevent the particle falling, which are measured. If the particle is spherical, there is no difficulty in relating the results obtained by different methods of size determination because the same diameter will be assigned to the sphere whatever the method of size determination adopted. But, in the case of an irregular particle it is not possible to assign a diameter (or a length, breadth and thickness) which will enable its volume or surface area to be accurately determined. The size dependent properties of the particles can still be determined and it is often convenient to represent the particle not by the measured property but by a 'size'. It is customary for the 'size' chosen to be the diameter of a sphere that exhibits the same property as the particle, that is, the sphere that has the same projected area or that just passes through the same sieve aperture or that falls at the same speed through a fluid. However, the sphere that is equivalent to a particle in regard to one size dependent property, say, projected area, is not necessarily of the same diameter as the sphere equivalent in regard to some other, property, say, free falling speed. An irregular particle is, therefore, assigned different sizes by different methods of determination and results obtained by different methods cannot be combined directly. Further, it is to be noted that the size-dependent properties of a non-spherical particle depend on its orientation so that its size even as determined by a single method is no longer a constant of the particle.

A-1.2 The discrepancy between the 'sizes' assigned to a particle by different methods depends on the departure of the particle from sphericity and the 'sizes' from different methods can be combined to give non-dimensional parameters known as shape factors, which serve to characterize the shape of the particle. A single shape factor does not characterize a particle exclusively and the particle has as many shape factors as there are pairs of methods of determining its size. Recommended factors are given below for use, in the absence of special knowledge, in correlating the following particle diameters:

- a) Sieve — Nominal sieve aperture through which the particle just passes.
- b) Projected — Diameter of circle of area equal to the projected area of the particle resting in its most stable position.

- c) Stokes — Diameter of sphere which has the same density and the same free falling velocity as the particle in a fluid under identical conditions within the range of Stokes' law.
- d) The conversion values are given below:

<i>Sl No.</i>	<i>To Convert</i>	<i>Multiply by</i>
1)	Sieve to projected	1.40
2)	Sieve to Stokes	0.94
3)	Projected to sieve	0.71
4)	Projected to Stokes	0.67
5)	Stokes to sieve	1.07
6)	Stokes to projected	1.50

Example:

Particles which just pass a 75-micron sieve have a mean projected diameter of 105 microns and a mean Stokes diameter of 70.5 microns.

A-1.3 These conversion factors should be applied with caution in case there is any suspicion that the particles of the powder under test are of extreme shapes, for example, if they have cleavage planes or are acicular. It is preferable to use then correlation factors which are specifically determined for the powder under test. Possible methods of establishing the factors are:

- to overlap the methods of size determination so that one or more size classes are assessed by both methods, and
- to size a sample of the material similar to the sample under test by both methods of size determination.

A-1.4 A numerical example is given in Table 9 to show how the results of a sieve analysis of a powder are combined with a microscope sizing of the fraction passing a 75-micron IS Sieve.

APPENDIX B

(Clause 3.2)

SIZING ACICULAR PARTICLES

B-1. DETAILS OF SIZING

B-1.1 The geometric mean of diameters (size obtained from microscopic area measurements) and the arithmetic mean of diameters (size obtained by unidirectional length measurements) tend to approach one another for increased number of measurements so long as the shape factor of the particles (ratio of the measured maximum to the measured minimum) does not exceed 4. The scope of the standard is, therefore, restricted to particles for which the length of the smallest rectangle enclosing the projected image does not exceed four times the breadth of the rectangle. Since the projected areas of particles of more elongated shape cannot be

TABLE 9 EXAMPLE OF COMBINATION OF SIEVE AND MICROSCOPE SIZINGS

(Clause A-1.4)

SIEVE ANALYSIS, SIEVE SIZE	MICROSCOPE ANALYSIS				COMBINED SIEVE MICROSCOPE ANALYSIS			
	Weight Passing Sieve as Percent of Total Sample	Projected Diameter	Sieve Dia- meter Eq- uivalent to Projected Diameter*	Weight Un- der Size as Percent of Weight Passing 75 Micron IS Sieve	Weight Un- der Size as Percent of Total Sam- ple	Equivalent Sieve Dia- meter	Percentage of Weight Un- dersize	Percentage of Weight Oversize
Microns	Percent	Microns	Microns	Percent	Percent	Microns	Percent	Percent
210	94	—	—	—	—	210	94	6
150	80	—	—	—	—	150	80	20
105	59	—	—	—	—	105	59	41
75	45	106	75	100	45	75	45	55
—	—	75	53	86	39	53	39	61
—	—	53	37	63	28	37	28	72
—	—	37	27	40	18	27	18	82
—	—	27	19	25	11	19	11	89
—	—	19	13	14	6	13	6	94

*Use multiplying factor of 0.71 from Appendix A.

reliably compared with those of circles, such particles should be assessed according to their lengths and breadths. The measurement may be made, by a ruler, on the projected images of the particles, the standard graticule grid or some similar rectangular grid being used to define the area of the sample fields. Alternatively a non-standard graticule of the type shown in Fig. 5 may be constructed. The graticule is ruled with reference to rectangles in place of circles, the shorter dimensions of these rectangles being compared with the breadths and the longer dimensions with the lengths of the particles. A suggested length to breadth ratio for the rectangles is 2:1.

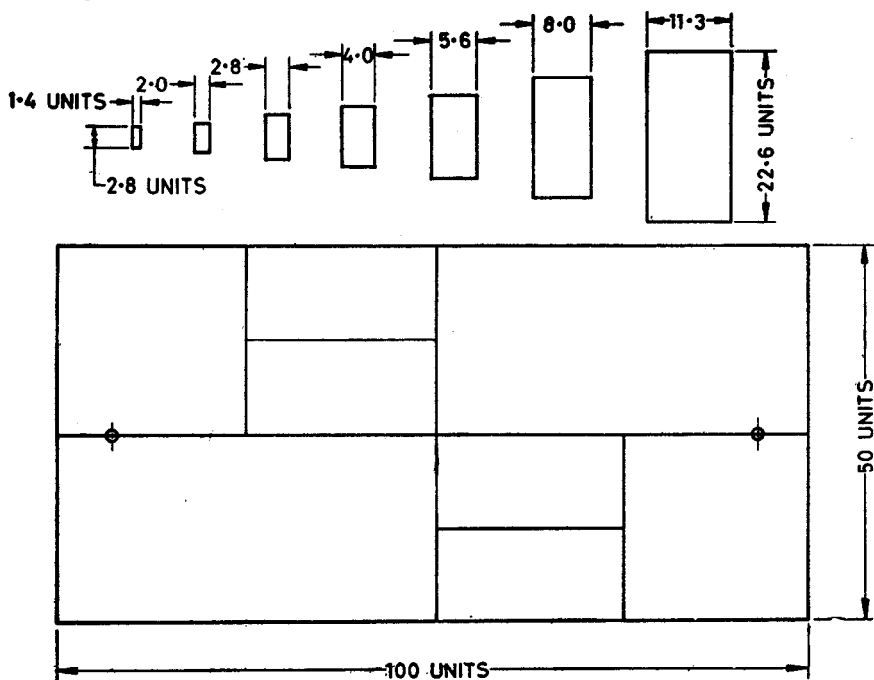


FIG. 5 NON-STANDARD GRATICULE FOR SIZING ACICULAR PARTICLES

B-1.2 The particles may be grouped into classes according to their breadths. A few full determinations on an acicular material may show that particles in the different classes may have the same average length to breadth ratio, thus making it possible in such cases for the weight distribution to be determined by measurement of only either the breadth or the length instead of both. If the powder consists of both spherical and acicular particles the two types of particles may be assessed separately by the appropriate method and from the results the relative percentages of spheres and needles may be calculated.

APPENDIX C*(Clause 4.2)***PROCEDURE FOR PREPARATION OF ANALYSIS SAMPLE****C-1. DETAILS OF PREPARATION**

C-1.1 Methods of preparing a sample of about 5 g in weight so that it is representative of the laboratory sample are given in IS:4879-1968*. A few milligrams of the material from this intermediate sample is incorporated in a viscous liquid (glycerol, glycerine jelly, medicinal paraffin etc) in which the powder under test has been previously found to disperse completely. The viscous shear acts to separate the flocculates without shattering the individual particles. Care shall be taken to prevent formation of air bubbles in the liquid while incorporating the powder into it. After satisfactory mixing, a drop of suspension is removed with a dropping rod and transferred to a clean microscope slide. A microscope cover slip is then gently lowered on to it. The cover slip should not be pressed but should be carefully slid to prevent selective removal of the larger particles to the edge of the cover slip. Also, lightly greasing the underside of the cover slip around the edge helps to prevent spreading of the liquid outside the edges of the cover slip and also evaporation of the suspending liquid for a long enough time for the count to be performed.

C-1.2 In case it is not possible to find a viscous liquid which may disperse the powder sufficiently well, it is necessary to use a more mobile liquid with the addition of a dispersing agent to prevent flocculation of the particles. The suspending liquid and dispersing agent will be specific to the powder under test, so that their choice is a matter of experiment and experience.

APPENDIX D*(Clauses 5.8 and 8.2)***PROCEDURE FOR ADJUSTMENT OF MICROSCOPE****D-1. DETAILED PROCEDURE**

D-1.1 All the elements of the optical system should be properly aligned and they should be focussed along the optical axis. Instructions for the adjustment of projection microscopes are usually supplied by the manufacturers.

D-1.2 The procedure for adjustment of bench microscopes is given below (see Fig. 1):

*Method of sub-division of gross sample of powder used for determination of particle size.

- a) Position the microscope with the body tube vertical and the stage horizontal.
- b) Centre the physical source of illumination (filament or arc) to lie on the optical axis of the lamp lens. Centre the lamp diaphragm with respect to the lamp lens. Place the source at a distance of 25 cm from the microscope mirror and adjust it so that the axis through the lamp diaphragm, lamp lens and the primary source passes through the centre of the mirror of the microscope. Coloured light is sometimes useful for obtaining contrast with strongly coloured materials. When a mercury arc is used it is extremely essential to use a filter to exclude ultra violet light in order to avoid undue irritation to the eye.
- c) Insert objective, eyepiece and substage condenser. Take the substage condenser close to its upper limit of travel and closing the substage condenser diaphragm tilt the mirror to focus an image of the physical source on the diaphragm, temporarily removing the diffusing screen of the lamp housing, if any. If the lamp lens cannot be moved for this purpose, move the whole lamp housing.
- d) Place the slide on the stage, open the substage condenser diaphragm and focus the objective on the particles. The position of focus is conveniently found by focussing first on the edge of the slide which can be found by moving the edge across the field of view while, at the same time, operating the fine focus adjustment. When immersion objective is used it should be oiled to the slide and to the substage condenser also, if the nominal numerical aperture of the objective exceeds 1.0. The oil used should have the refractive index stipulated by the manufacturer of the objective and condenser (usually either 1.515 or 1.524). The objective should be brought slowly into contact with the oil to avoid trapping air bubbles.
- e) With the particles in focus, close the lamp iris partially and focus edges by racking the substage condenser up or down. Then, adjust the mirror so that the lamp iris is central in the field of view. The lamp iris will have to be closed to its smallest possible opening with high power objectives.
- f) Open the lamp diaphragm and close the substage condenser diaphragm partially. Replace the eyepiece with a pin hole cap without disturbing the focus. Place a ground glass diffusing screen immediately below the substage condenser. Bring the image of the substage diaphragm as seen through the pin whole cap concentric with the aperture stop of the objective, by means of the substage centring screws. Remove the diffusing screen and replace the eyepiece. Recentre the image of the lamp iris in the field of view as indicated in (e).

- g) Remove the eyepiece, unscrew the eyepiece lens system (positive eyepiece) or the eye lens (negative eyepiece) and insert the graticule so that it rests on the field stop (*see* Fig. 1). The eye lens should be screwed back until the graticule is in focus. The position of the focus which depends on the accommodation of the eye of observer is adjusted by alternatively looking at a distance object and through the eyepiece until, by moving the eye lens, the distant object and graticule are seen clearly without changing the accommodation of the eye.
- h) Replace the eyepiece with its graticule in the draw tube of the microscope. Adjust the length of the microscope to the setting established for the particular objective and eyepiece as indicated in Table 6. Re-focus the particles and substage condenser, if necessary. Adjust the intensity and colour of illumination by means of the neutral and coloured filters. The voltage of the light source should not be altered for changing the intensity as by so doing the colour of the light also will change. The level of illumination should be low in order to reduce fatigue to the eye.

APPENDIX E

(*Clauses 7.2, 7.5 and 7.5.1*)

CHOICE OF GRID LENGTH OF EYEPIECE GRATICULE

E-1. CRITERIA FOR CHOICE

E-1.1 Select the objectives required to cover the size range of particles present on the slide. Select the eyepiece (of power $\times 20$ or more for the bench microscope) which is to be used for counting and sizing and insert into it a suitable micrometer scale. Determine for each objective the magnification, from the stage to the plane of the eyepiece graticule, for the shortest and the longest tube length adjustment available on the microscope. Divide the measured magnification by the relative magnification of the objectives given in Table 7 according to their focal lengths. Note the largest value obtained at the shortest tube length and the smallest value obtained at the longest tube length. Multiply both values by 0.937. Select an eyepiece graticule with a grid length between the values, as illustrated in the example below. Confirm that the graticule having this size of grid can be employed with the eyepiece chosen. The length of grid shall not exceed five-eighths of the diameter of the field stop of the eyepiece.

Example:

The measured magnifications at the two extreme tube lengths in the case of 4 objectives used are given in col 2 and 3 of Table 10. The ratios of measured to relative magnification are given in col 5 and 6 of the table. The same eyepiece $\times 20$ was used with each objective.

TABLE 10 EXAMPLE OF CHOICE OF GRID LENGTH FOR AN EYEPIECE GRATICULE

FOCAL LENGTH	MEASURED MAGNIFICATION AT PLANE OF GRATICULE FOR		RELATIVE MAGNIFICATION FROM TABLE 7	RATIO OF MEASURED TO RELATIVE MAGNIFICATION AT	
	Shortest Tube Length	Longest Tube Length		Shortest Tube Length	Longest Tube Length
mm					
(1)	(2)	(3)	(4)	(5)	(6)
31.6	4.24	5.88	1	4.24	5.88
16	12.05	15.38	$2\sqrt{2}$	4.26	5.43
4.5	47.62	62.5	$8\sqrt{2}$	4.21	5.53
1.9	111.11	142.86	$16\sqrt{2}$	4.91	6.32

The largest value at the shortest tube length is 4.91 (for 1.9 mm objective) and the smallest value at the longest tube length is 5.43 (for 16 mm objective). Multiplication by 0.937 gives the two values 4.60 and 5.09. A graticule of grid length 4.75 mm lies within the limits. A graticule of grid length up to 5 mm can be used. The diameter of the field stop of the eyepiece lens is 10 mm and the eyepiece could accommodate a graticule of grid length up to 6.25 mm.

APPENDIX F

(Clause 9.2.1)

WORKED EXAMPLE OF DETERMINATION OF NUMBER SIZE DISTRIBUTION

F-1. DETAILS OF WORKED OUT EXAMPLE

F-1.1 It is considerably easier to determine the size distribution by number since the same total sample area is used for all size classes and also the minimum number of particles that is necessary to count, namely 625, is specified in advance. A preliminary examination of the sample slide is made to ensure that the particle concentration and the size of the sample field are such that at least 96 sample fields are examined for the control size class and at least 12 preferably 24 for any other size class.

F-1.1.1 It is recommended that in counting at the highest magnification those particles larger than circle 7 should be recorded as such, (although

this figure is not used in the calculation of number distribution) and these are subsequently assessed at the next lower magnification. These two estimates serve as a useful check over counting.

F-1.1.2 Also, it is recommended that with the beginning of the counting at the highest magnification an initial 25 fields be examined such that each field equals to the area covered by the graticule grid. These fields should be spaced regularly over the whole area occupied by particles. On the basis of the total number of particles, including those larger than circle 7 counted in these 25 fields, an estimate is made of the number of further fields that need to be examined to bring the number of particles counted up to 625.

F-1.1.3 A worked out example to show the method of calculation of a size distribution by number is given in Table 11.

APPENDIX G

(Clauses 9.3.1, 14.1.4 and 14.1.6)

WORKED EXAMPLE OF DETERMINATION OF WEIGHT SIZE DISTRIBUTION

G-1. PROCEDURE

G-1.1 In the determination of size distribution by weight the total sample area to be examined varies from class to class and may not be specified in advance. The counting procedure has to be controlled by various factors calculated at different stages of its determination. The following scheme is recommended in the determination of weight size distribution:

- a) *Preliminary Examination* — A preliminary examination of a scan (of width equal to that of the graticule grid and 10-20 mm in length) across the middle of the area occupied by the particles on the sample slide is made with the microscope set at the lowest magnification required. The total number of particles in the top three classes occurring in the sample is recorded. From this the number of such scans required to give a total of about 150 particles in the top three classes is estimated. If this estimated number is less than 5, calculate a reduced length of scan to increase it to at least 5. The count recorded in the preliminary scan is not included in the analysis proper.
- b) *Analysis at Lowest Magnification* — The number of particles counted in the top three classes are recorded from the required number of scans as decided in (a) distributing them evenly over the whole area occupied by the particles. On the basis of these figures an estimate is made to get the total sample area expected to contain 25 particles

TABLE 11 ILLUSTRATIVE EXAMPLE OF CALCULATION OF SIZE DISTRIBUTION BY NUMBER
(Clause F-1.1.3)

OBJECTIVE $f/$ (NUMERICAL APER- TURE)	CIRCLE NUMBERS	SIZE CLASS LIMITS	AREA OF THE SAMPLE FIELD (a_r)	NUMBER OF SAMPLE FIELDS (n_r)	TOTAL SAMPLE AREA ($n_r a_r$)	NUMBER OF PARTI- CLES COUNTED IN CLASS (m_r)	NUMBER OF CONC- ENTRA- TION IN CLASS ($N_r = \frac{m_r}{n_r a_r}$)	NUMBER PERCENT IN CLASS ($P_r = \frac{100N_r}{\Sigma N_r}$)	NUMBER OF SIZE DISTRIBUTION		STANDARD ERROR PERCENT ($S(P_r) = \sqrt{\frac{P_r (100 - P_r)}{\Sigma (m_r)}}$)							
									Size	Percent (Under- size)								
(1)	(2)	(3) (microns)	(4) mm ²	(5)	(6) mm ²	(7)	(8) (per mm ²)	(9) (per mm ²)	(10) (microns)	(11)	(12)							
16.3 mm (0.25)	{	>7	>37	0.054 875	$\frac{1}{4} \times 24$	0	0	0	37	100.0	0.59							
		7-6	37-27									19	115	2.6	27	97.4	0.65	
		6-5	27-19									23	140	3.2	19	94.2	0.75	
		5-4	19-13									31	188	4.3	13	89.9	0.84	
		4-3	13-9.4									39	237	5.4	9.4	84.5	0.90	
4.5 mm (0.65)	{	7-6	9.4-6.6	0.003 429 7	48	0.164 6	46	279	6.3	6.6	78.2	0.90						
		6-5	6.6-4.7										52	316	7.2	4.7	71.0	0.96
		7-6	4.7-3.3										58	352	8.0	3.3	63.0	1.01
1.9 mm (1.3)	{	6-5	3.3-2.3	0.000 857 42	192	0.164 6	63	383	8.7	2.3	54.3	1.05						
		5-4	2.3-1.7*				72	437	9.9	1.7	44.4	1.11						
		4-3	1.7-1.2				81	492	11.2	1.2	33.2	1.17						
		3-2	1.2-0.8				94	571	13.0	0.8	20.2	1.25						
		2-1	0.8-0.6*				146	887	20.2	0.6	0	1.49						
Column totals						$\Sigma m_r = 724$	$\Sigma N_r = 4\ 397$	100.0										

*Control size class.

Standard error of control size class due to sampling = $S(P_o) = \sqrt{\frac{20.2 \times 79.8}{724}} = 1.49$ percent [see 10.2]

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of the top class. Similarly on the above basis the total sample areas necessary for the second and third classes are estimated. After this estimation further necessary counting is carried out distributing the areas regularly. Calculate the accuracy factors F to ensure that those of the second and third classes are smaller than F_1 . If it is found subsequently that more than 10 percent by weight of the material is in the top class, 25 particles in the top class will not have produced the desired accuracy and further counting is necessary.

- c) The magnification is increased by, say, a factor 2 and the areas to be counted to get the desired accuracy for another 3 classes are assessed. For this purpose 25 fields are examined initially using the whole grid area for the 3 classes (or the whole grid area for the largest class and one-half of the grid area for the other two classes) without changing the magnification. On the basis of the numbers N_r recorded in these counts decide what further area needs to be examined for each class. After completion of the counting of the minimum number of required areas for each class, calculate F_r for each class to ensure that it is less than F_1 . Similar procedure is repeated by changing to the next higher magnification for other size classes.
- d) When the accuracy factor F_r for each size class has been reduced below F_1 calculate the weight size distribution (*see 9.3*). Check that the standard error due to sampling of the percentage weight in the control size class is less than 2 percent.
- e) The process is illustrated by the worked example given under **G-2**.

G-2. WORKED EXAMPLE

G-2.1 On preliminary examination it is found that 5 scans will yield about 150 particles in the top three size classes occurring in the sample.

Initial number of scans at lowest

magnification = 5

Total area inspected in initial
scans

$$= 5 \times 10 \times 0.439 \\ = 21.95 \text{ mm}^2$$

Number of particles recorded:

106 – 75 microns	12
75 – 53 microns	56
53 – 37 microns	169

The total number of particles observed are more than 150 to satisfy the condition in (a) above.

G-2.2 Calculation of Further Scanning Area Required for These Top Three Classes

Mean diameter of the top class	$= d_1$
Mean diameter of the second class	$= d_2$
Mean diameter of the third class	$= d_3$
Number concentration in top class	$= \frac{12}{21.95}$ $= 0.547/\text{mm}^2$ $= N_1$

To ensure that the standard error due to sampling is not greater than 2 percent 25 particles of the top class should always be observed (see 14.1).

Therefore, estimated sample area	$= \frac{21.95 \times 25}{12}$ $= 45.7 \text{ mm}^2$ $= n_1 a_1$
Number of scans required	$= \frac{5 \times 45.7}{21.95}$ $= 10.4$, say 11 scans (of which 5 have already been carried out)

Similarly for the second class:

Number concentration	$= \frac{56}{21.95}$ $= 2.551/\text{mm}^2$ $= N_2$
$\frac{\text{Mean diameter of second class}}{\text{Mean diameter of top class}}$	$= \frac{d_2}{d_1} = \frac{1}{\sqrt{2}}$

Then from 14.1 the area ($n_2 a_2$) to be examined for this class to ensure that standard error due to sampling does not exceed 2 percent

$$\begin{aligned}
 &= n_1 a_1 \left(\frac{d_2}{d_1} \right)^2 \frac{N_2}{N_1} \\
 &= 45.7 \left(\frac{1}{\sqrt{2}} \right)^2 \frac{2.551}{0.547} = 26.64 \text{ mm}^2
 \end{aligned}$$

$$\begin{aligned}\text{Number of scans required} &= \frac{5 \times 26.64}{21.95} \\ &= 6.1, \text{ say } 7 \text{ scans.}\end{aligned}$$

For the third class:

$$\begin{aligned}\text{Number concentration} &= \frac{169}{21.95} = 7.699/\text{mm}^3 \\ &= N_3\end{aligned}$$

$$\frac{\text{Mean diameter of third class}}{\text{Mean diameter of top class}} = \frac{d_3}{d_1} = \frac{1}{2}$$

Area to be examined to ensure the desired accuracy

$$\begin{aligned}&= n_1 a_1 \left(\frac{d_3}{d_1} \right)^6 \frac{N_3}{N_1} \\ &= 45.7 \left(\frac{1}{2} \right)^6 \frac{7.699}{0.547} \\ &= 10.1 \text{ mm}^2\end{aligned}$$

$$\text{Number of scans required} = \frac{5 \times 10.1}{21.95} = 2.3, \text{ say } 3 \text{ scans}$$

The count might therefore be carried out over a further minimum of 6 scans for the first class, 2 scans for the second class and no more for the third class.

G-2.3 Calculation of Accuracy Factors — In the actual count as recommended in G-1.1 the following results were obtained.

$$106 - 75 \text{ microns } 51 \text{ particles } (m_1) \text{ in } (0.439 \times 10 \times 20) \\ = 87.8 \text{ mm}^2$$

$$75 - 53 \text{ microns } 140 \text{ particles } (m_2) \text{ in } (0.439 \times 10 \times 12) \\ = 52.7 \text{ mm}^2$$

$$53 - 37 \text{ microns } 169 \text{ particles } (m_3) \text{ in } (0.439 \times 10 \times 5) \\ = 22.0 \text{ mm}^2$$

G-2.3.1 The corresponding number concentrations N_1 , N_2 and N_3 are therefore 0.581, 2.66 and 7.70/mm³ respectively.

G-2.3.2 The weighting factors d_1^3 , d_2^3 , and d_3^3 are obtained from Table 1 and are 741 000, 262 000, and 92 700.

The accuracy factors are calculated according to the equation.

$$F_r = \frac{N_r d_r^3}{\sqrt{m_r}}$$

$$\text{Therefore } F_1 = \frac{0.581 \times 741\,000}{\sqrt{51}} = 60\,285$$

= the control factor F_0

$$F_2 = \frac{2.66 \times 262\,000}{\sqrt{140}} = 28\,911$$

$$F_3 = \frac{7.70 \times 92\,700}{\sqrt{169}} = 54\,911$$

G-2.3.3 Since F_2 and F_3 are both less than F_1 sufficient particles have been counted in the second and third classes to ensure the desired accuracy. The process is repeated at the three higher magnifications and the weight size distribution calculated as shown in Table 12.

TABLE 12 ILLUSTRATIVE EXAMPLE OF CALCULATION OF SIZE DISTRIBUTION BY WEIGHT

(Clause G-2.3.3)

OBJECTIVE f / (NUMERICAL APER- TURE)	CIRCLE NUM- BERS	SIZE CLASS LIMITS	AREA OF SAM- PLE FIELDS (a_r)	NUMBER OF SAM- PLE FIELDS (n_r)	TOTAL SAMPLE AREA ($n_r a_r$)	NUMBER OF PAR- TICLES COUNTED IN CLASS (m_r)	NUMBER CON- CENTRA- TION ($N_r = \frac{m_r}{n_r a_r}$)	WEIGHT- ING FACTOR FROM TABLE 1 (d_r^3)	RELA- TIVE WEIGHT IN CLASS ($N_r d_r^3$)	WEIGHT PERCENT IN CLASS ($Q_r = \frac{100 N_r d_r^3}{\Sigma (N_r d_r^3)}$)	ACCURACY FACTOR FOR CLASS ($F_r = \frac{N_r d_r^3}{\sqrt{m_r}}$)	WEIGHT SIZE DISTRIBUTION		STANDARD ERROR PERCENT	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	
		microns	mm ²		mm ²		per mm ²					microns			
31.6 mm (0.10)	{	7 - 6	106 - 75*	0.439 × 10 × 20	200	87.8	51	0.581	741 000	430 521	14.0	60 285	106	100.00	1.66
		6 - 5	75 - 53	0.439 × 10 × 12	120	52.7	140	2.66	262 000	696 920	22.6	58 911	75	86.00	1.41
		5 - 4	53 - 37	0.439 × 10 × 5	50	22.0	169	7.70	92 700	713 790	23.2	54 907	53	63.40	1.31
16.3 mm (0.25)	{	7 - 6	37 - 27	0.054 875	100	5.49	76	13.8	32 800	452 640	14.7	51 763	27	25.50	1.42
		6 - 5	27 - 19	„	50	2.74	83	30.3	11 600	351 480	11.4	38 580	19	14.10	1.10
		5 - 4	19 - 13	„	50	2.74	137	50.0	4 100	205 000	6.7	17 521	13	7.40	0.53
		4 - 3	13 - 9.4	„	25	1.37	124	90.5	1 450	131 225	4.3	11 780	9.4	3.10	0.37
4.5 mm (0.65)	{	7 - 6	9.4 - 6.6	0.003 429 7	50	0.171	17	99.4	512	50 893	1.7	12 344	6.6	1.40	0.41
		6 - 5	6.6 - 4.7	„	50	0.171	26	152	181	27 512	0.9	5 396	4.7	0.50	0.17
1.9 mm (1.3)	{	7 - 6	4.7 - 3.3	0.000 857 42	25	0.021 4	34	159	64	10 176	0.3	1 745	3.3	0.20	0.05
		6 - 5	3.3 - 2.3	„	25	0.021 4	41	192	22.7	4 358	0.1	681	2.3	0.10	0.02
		5 - 4	2.3 - 1.7	„	25	0.021 4	47	220	8.00	1 760	0.06	257	1.7	0.04	0.01
		4 - 3	1.7 - 1.2	„	25	0.021 4	52	243	2.83	688	0.02	95	1.2	0.02	0.003
		3 - 2	1.2 - 0.8	„	25	0.021 4	56	262	1.00	262	0.01	35	0.8	0.01	0.001
Column totals						$\Sigma m_r = 1\,063$	$\Sigma N_r = 1\,523$		$\Sigma N_r d_r^3 = 3\,077\,225$	99.99					

*Control size class.

Standard error of control size class due to sampling $S(Q_o) = \frac{14.0}{\sqrt{51}} \sqrt{1 - \frac{14.0}{50}} = 1.66$ percent.

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